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# RESEARCH REPORT

RADIATION EFFECTS ON LIQUID PROPELLANTS

to

NATIONAL AERONAUTICS  
AND SPACE ADMINISTRATION



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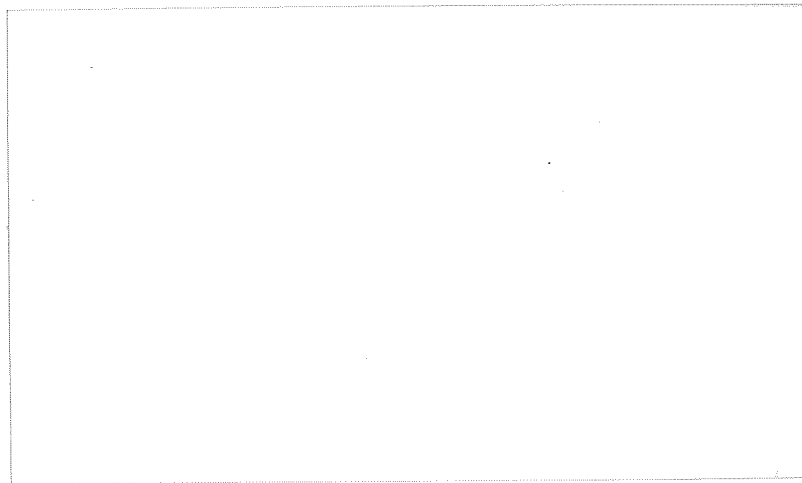
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FINAL REPORT

on

RADIATION EFFECTS ON LIQUID PROPELLANTS

to

NATIONAL AERONAUTICS  
AND SPACE ADMINISTRATION

January 22, 1971

NAS7-722

by

J. F. Kircher and D. A. Vaughan

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Pasadena, California 91103

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BATTELLE  
Columbus Laboratories  
505 King Avenue  
Columbus, Ohio 43201

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ABSTRACT

The effects of radiation on the stability and compatibility of hydrazine ( $N_2H_4$ ) and hydrazine-24% hydrazine nitrate ( $N_2H_4-24N_2H_5NO_3$ ) in four commercial-alloy container materials (1100Al, 6061Al T-6, 347 stainless steel, and Ti-6Al4V) were investigated. Radiolytic decomposition of the propellants gave G-values of about 4.5 and 7.6 for  $N_2H_4$  and  $N_2H_4-24N_2H_5NO_3$ , respectively, although it varied somewhat with container material and radiation dose. The  $N_2H_4$  was found to be compatible with all four alloys, whereas the  $N_2H_4-24N_2H_5NO_3$  reacted with all the materials, but reacted most with 347 SS. Although the magnitude of the reaction does not alter appreciably the surface structure of the capsule or of coupon test specimens, it is sufficient to contaminate the propellant and cause discoloration, and might clog the 347 SS capillary tubing of the feed system of a liquid propulsion system. A filter unit designed for fuel cleaning operated without plugging or visible attack by either propellant, but the ethylene-propylene O-ring seal showed evidence of crosslinking when irradiated under load.

# RADIATION EFFECTS ON LIQUID PROPELLANTS

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## INTRODUCTION

This program (Phase II of Order No. NAS7-722) supplements the Phase I research on radiation effects on liquid propellants (reported in Document CR109767\*) with additional data on selected propellants and materials unique to liquid-propulsion systems for space probes. The specific tasks of Phase II were (1) to evaluate the radiation effects on monopropellants, (2) to evaluate possible stress corrosion and the general compatibility of various candidate materials (used for containers, capillary tubing, and filters) with the propellants, and (3) to examine the reaction of materials employed in Phase I with fuels and oxidizers. Thus, Phase II was an extension of Phase I to other propellants and included additional candidate materials for liquid-propulsion systems. As the particular space-probe mission is expected to require long-term storability (up to 10 years) of the propellant, data on the physiochemical compatibility of it with the storage and the feed systems are needed for reasonable assurance of successful operation upon demand. In addition to exposure to the liquid propellant, the materials selected will be exposed to radiation from isotope or nuclear auxiliary power sources on the space probe, therefore the compatibility studies were conducted in and out of a radiation field. Wherever possible, data in this report are presented in engineering units to facilitate their use in design. The short duration of this program prohibited extrapolation to expected mission life, but the results did provide a basis for differentiating the behavior of propellants in four commercial materials.

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\* "Radiation Effects on Liquid Propellants", CR109767, J. F. Kircher, R. E. Best, J. E. Rollins, Feb. 2, 1970.

## EXPERIMENTAL PROCEDURE

### General Method

The effects of radiation on the selected liquid propellants were investigated by analysis of both the pressure buildup and the composition of the 78°C noncondensable gases before and after exposure to gamma radiation from a  $^{60}\text{Co}$  source. Several  $^{60}\text{Co}$  radiation sources were assembled and dosimetry measurements were performed on each. A standard ferrous sulfate dosimeter was used. The dosimetry measurements were carried out in a physical setup identical with that which was used in the experiments described in Document CR109767.

Cobalt-60 gamma radiation was used throughout the experiments since radiation-induced decomposition is, in general, independent of the nature of the radiation and is a function only of the amount of energy deposited in the fuel materials. Also, in an on-board situation, the major radiation to which a fuel would be exposed may well be bremsstrahlung from the RTG, which is electromagnetic radiation of the same nature as gamma rays. The deleterious reactions occurring in the fuel systems generally result from free radicals, atoms, and excited molecules produced by the radiation. The reactions initiated by such chemically active species, however, are independent of the mode of formation of the initiating species. That is, the reaction of a free radical, for instance, in the fuel does not generally depend on the way in which the free radical was formed. Thus, the results generated in the research reported here should be generally applicable, regardless of the nature of RTG in an actual flight situation.

In one type of experiment, the propellants were contained in a glass vessel with and without test specimens of four commercial alloys (1100 Al, 6061 Al347 stainless steel, and Ti-6Al-4V) as shown in Figure 1. The test specimens consisted of U-bends (Figure 2) held under load by a notched bar of the same material. One specimen of each alloy was exposed to the liquid and one of each was exposed to the vapors of the propellant;

each specimen was separated from the adjacent ones by a glass disk. Details of specimen composition, preparation, and examination are presented in the following section of this report. Two vessels for each system were prepared--one for room storage and the other for exposure to radiation. This duplication was repeated for the second type of experiment, in which ~12-ml capsules (Figure 3) of the four alloys, obtained from Hoke, Inc., were filled with 5 ml of propellant for radiation-effects investigations.

All vessels and capsules were filled under an argon blanket, cooled to  $-78^{\circ}\text{C}$ , vacuum pumped to a pressure of 10 microns, and sealed before being warmed to room temperature for radiation exposure or storage. Pressure changes in the capsules were measured as a function of time and exposure to radiation to determine the amount of corrosion-produced gas or the extent of radiolytic decomposition. The pressure change during room storage of capsules containing the propellant was attributed to corrosion-produced gases. During exposure of the alloys to the propellants in glass vessels, the surfaces of the specimens were examined at low magnification (5X) for evidence of surface reactions. Upon completion of exposure, the test specimens and the capsules were examined metallographically and by electron diffraction to determine changes in surface structure and composition.

The 347-SS capillary tubing specified by the Sponsor and filter supplied by the Sponsor to be used in the Sponsor's liquid propulsion system were examined for flow characteristics and compatibility with the propellants before and after exposure to radiation and room storage. These materials were filled and immersed in the propellants contained in glass vessels during exposure.

#### Materials Specification and Preparation

The propellants selected for this investigation were a hydrazine-hydrazine nitrate solution ( $\text{N}_2\text{H}_4\text{-}24\text{N}_2\text{H}_5\text{NO}_3\text{-}1\text{H}_2\text{O}$ ) and a hydrazine-hydrazine azide solution ( $\text{N}_2\text{H}_4\text{-N}_2\text{H}_5\text{N}_3$ ), which were to be compared with hydrazine ( $\text{N}_2\text{H}_4$ ). Because attempts to obtain the  $\text{N}_2\text{H}_4\text{-N}_2\text{H}_5\text{N}_3$  solution were unsuccessful, the research effort was limited to investigation of the remaining two



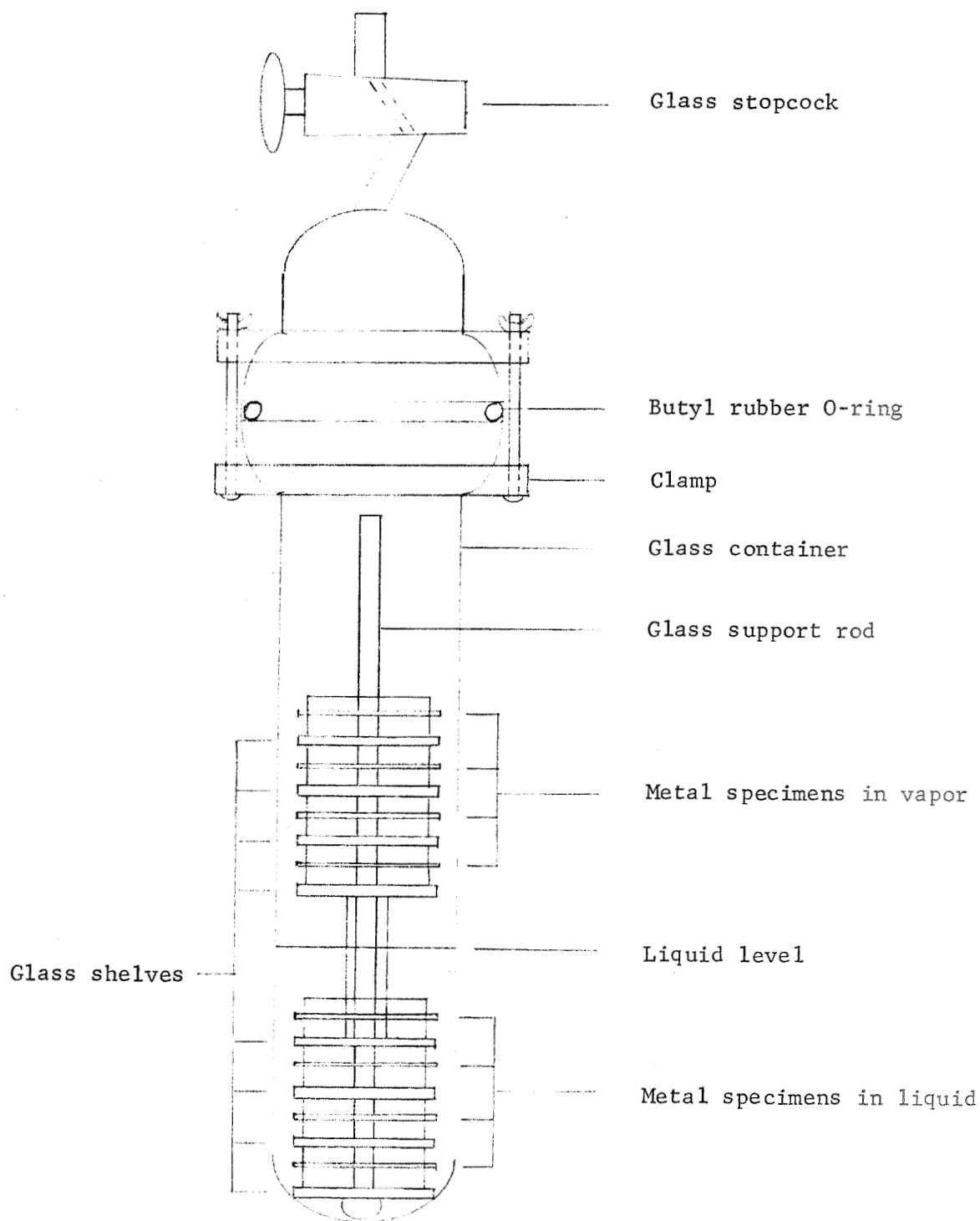


FIGURE 1. PYREX GLASS VESSEL (1/2 SIZE) AND SPECIMEN POSITION FOR EXPOSURE OF ALLOYS TO PROPELLANT LIQUID AND VAPORS

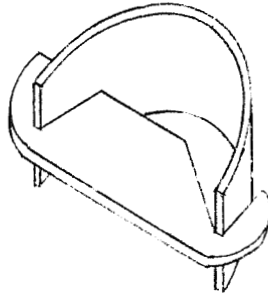


FIGURE 2. U-BEND TEST SPECIMEN  
(~ FULL SIZE)

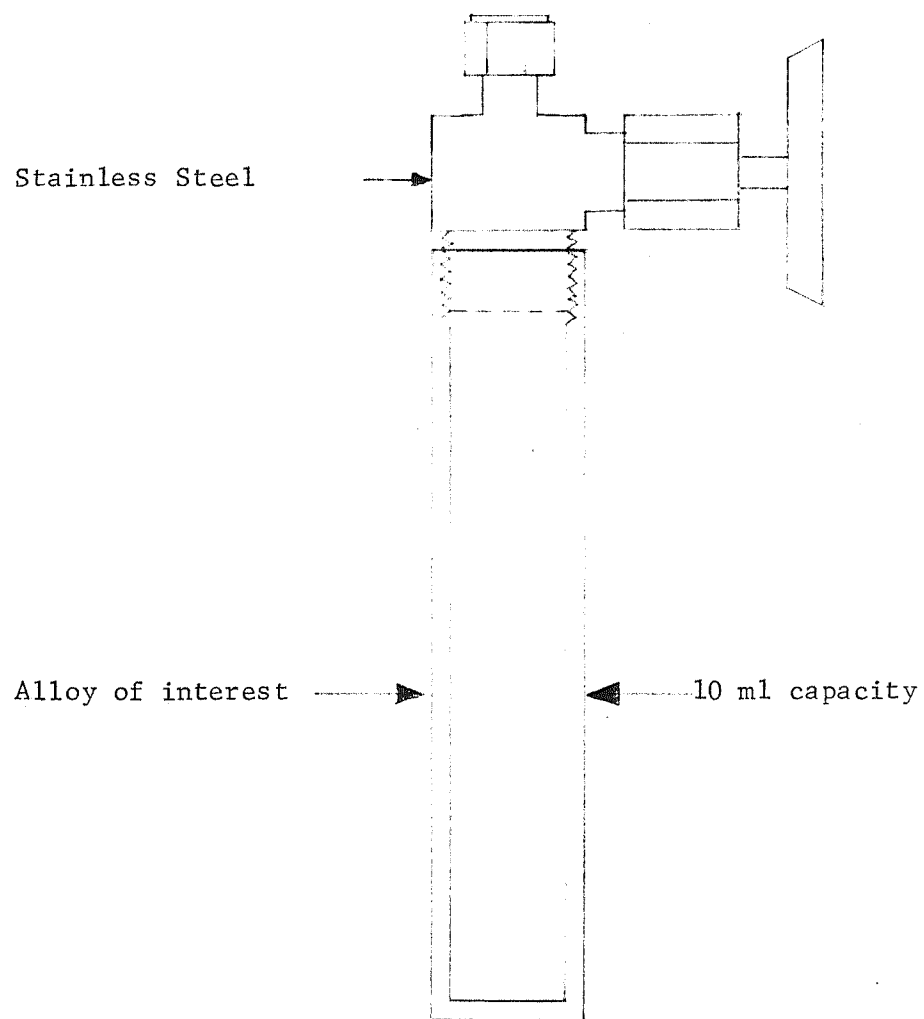


FIGURE 3. CAPSULE AND VALVE FOR CONTAINING PROPELLANT IN COMPATIBILITY AND RADIOLYTIC DECOMPOSITION STUDIES

materials. The  $N_2H_4$  was obtained from Olin Chemicals as a clear liquid with a few white, fibrous-type particles that tended to settle out on standing. Attempts to remove these fibers were not completely successful, but the fibers were avoided by decanting the liquid used for the experiments. The  $N_2H_4$ - $24N_2H_5NO_3$  was prepared at Battelle by the method supplied by S. P. Vango of JPL, which is described in the Appendix. These materials were handled in an argon-filled glove box or in evacuated and sealed containers.

As indicated earlier, four commercial alloys were candidates for the storage and feed systems, providing they exhibited compatibility with the propellants. These materials are listed below, with compositional data for the two types of samples used in this program.

<u>Alloy</u>	<u>Form</u>	<u>Composition</u>
1100 Al	Capsule	Al + (Fe+Si-1.0, Cu-0.20, Zn-0.10, Mn-0.05, other -0.15)
	63-mil sheet	(same specification)
6061 Al T-6	Capsule	Al + (Fe-0.70, Si-0.80, Cu-0.40, Zn-0.25, Mn-0.15, Mg-1.20, Cr-0.35, Ti-0.15, other -0.15)
	51-mil sheet	Al + (Fe-0.40, Si-0.61, Cu-0.26 - Mn-0.20, Mg-0.80, Cr-0.19, Ti-0.05, other -0.1)
347 SS	Capsule	Fe + (Cr-17.83, Ni-10.50, Mn-1.56, Si-0.66, Mo-0.32, Cu-0.24, Cb-0.73, Co-0.10, Ta-0.01, P-0.027, S-0.025)
	32.5-mil sheet	Fe + Cr-17.85, Ni-9.45, Mn-0.94, Si-0.70, Cb+Ti-0.66, C-0.057, P-0.029, S-0.008)
Ti-6Al-4V	Capsule	Ti + (Al-6.3, V-4.2 Fe-0.13, C-0.022, N-0.011, O-0.15, H-0.006)
	63-mil sheet	Ti + (Al-5.9, V-4.1, Fe-0.15, C-0.023, N-0.014, O-0.11, H-0.008)

All capsules were prepared by Hoke, Inc., by drilling out 3/4-inch rod material to provide a cavity of ~ 10 ml beneath the tapped end to which a stainless steel valve was attached. Inspection at Battelle revealed that the inner wall of the valve stems and the capsule cavities were contaminated. Therefore, all valves were removed and cleaned by the methods used for the U-bend specimens, reassembled, and vacuum tested before loading for radiation-effect or storage tests.

U-bend coupons of the sheet materials were made by bending flat stock, 1/2 inch by 2 inches, to fit into notches 1-1/2 inches apart in a flat strip of the same material. Each coupon and its holding clip was solvent-cleaned and chemically or electrochemically polished, as indicated below, prior to mounting for exposure to the propellants.

<u>Material</u>	<u>Polish</u>
Aluminum alloy	Immersed 10 minutes in 50°C solution of 90 ml $H_2O$ + 10 ml $H_3PO_4$
Titanium	Immersed 1 minute in 0°C solution of 80 ml lactic acid + 20 ml $HNO_3$ + 20 ml HF
Stainless steel	Anodic 20 V for ~ 3 minutes in an electrolyte of 60 ml $H_3PO_4$ + 40 ml $H_2SO_4$ with the solution cooled in a water bath.

All pieces were rinsed thoroughly in water, then in alcohol, and were dried in a warm-air blast. Each coupon was examined metallographically for surface structure prior to exposure to determine whether the surfaces were clean and polished without pits or deep etching. Duplicates of the flat pieces were prepared and stored in closed vials for subsequent comparison with the exposed specimens.

## EXPERIMENTAL RESULTS

### Evaluation of Radiation Effects on Propellants

#### Radiolytic Decomposition

Irradiation of the propellants in closed containers by exposure to 0.53 megarad per hour of gamma radiation resulted in an increase in the gas pressure, which was measured periodically and converted to moles of gas produced per gram of liquid irradiated. This gas, of course, contained the gas produced by reaction of the propellant with the container materials in addition to the products of radiolytic decomposition. Total gas production is presented in Section A of Tables 1 and 2, which show considerable variation with container material, particularly that produced by the  $\text{N}_2\text{H}_4$ - $24\text{N}_2\text{H}_5\text{NO}_3$  propellant. As these variations were believed to be due to differences in corrosion behavior of the container materials, the increase in gas pressure after storage of irradiated and nonirradiated capsules was measured (see Sections B and C of Tables 1 and 2). The volume of gas generated by unirradiated  $\text{N}_2\text{H}_4$  was very low, while significant quantities were produced (appreciably more after irradiation) when the  $\text{N}_2\text{H}_4$ - $24\text{N}_2\text{H}_5\text{NO}_3$  was contained in 347-SS. Correcting the total gas generation by subtracting the volume of corrosion-generated gas from the total volume generated indicated the amount of propellant radiolytically decomposed. Section D, Tables 1 and 2 show that the quantity of radiolytic decomposition depends upon fuel and radiation dose, as would be expected.

The data presented in Tables 1 and 2 suggest two general conclusions. First, there is undoubtedly some relation between the radiation stability of the two fuels investigated and their overall stability or storability, which is dependent on the container material. Second, decomposition, once initiated by radiation, persists for some time even after the irradiation ceases. In the case of neat hydrazine, the post irradiation decomposition as indicated by pressure buildup seems to tend toward a value that is not

TABLE 1. DECOMPOSITION OF HYDRAZINE BY RADIATION AND CORROSION

Total Radiation, Mrads	Time in Capsules, hr	Alloy Type				
		1100 Al	6061 Al	347 SS	Ti-6Al-4V	
<u>A. Total Gas Buildup,</u> <u>Mole x 10<sup>6</sup> per Gram of Liquid</u>						
0.5	71	3.15	3.95	3.76	3.60	
1.6	73	7.72	9.90	9.15	8.65	
11.7	96	52.5	65.7	51.6	59.6	
22.8	148	102.5	121.1	100.5	119.7	
33.4	172	149.8	175.6	146.1	173.8	
44.5	288	191.7	234.0	192.3	230.6	
55.0	312	211.1	--	246.4	287.2	
<u>B. Gas Buildup With Time After Exposure</u> <u>to Radiation Dose Indicated</u>						
11.7	24	1.4	2.6	3.0	1.3	
33.4	66	3.1	2.6	3.1	3.7	
55.0	144	3.1	1.0	3.6	3.1	
Avg/hr		0.041	0.054	0.066	0.043	
<u>C. Gas Buildup With No Irradiation</u> <u>(Corrosion Gas)</u>						
0	264	1.9	1.8	1.4	0.7	
0	980	3.2	5.3	6.8	4.8	
Avg/hr		0.005	0.006	0.006	0.004	
<u>D. Radiolytic Produced Gas</u> <u>(Total - Corrosion Gas)</u>						
					<u>Avg</u>	
0.5		2.80	3.52	3.33	3.32	3.24
1.6		7.36	9.46	8.71	8.36	8.47
11.7		51.2	64.0	49.6	58.3	55.8
22.8		99.1	116.6	97.0	116.2	107.2
33.4		145.4	169.8	139.1	169.3	155.9
44.5		182.5	221.9	177.1	221.1	200.8
55.0		200.9	--	230.2	276.6	235.9
<u>E. G-Values (Molecules of</u> <u>Gas/100 ev Absorbed)</u>						
0.5		5.4	6.8	6.4	6.4	6.3 (a)
1.6		4.4	5.7	5.2	5.0	5.1
11.7		4.3	5.3	4.1	4.8	4.6
22.8		4.2	5.0	4.1	4.9	4.6
33.4		4.2	4.8	4.0	4.8	4.5
44.5		4.2	4.8	3.8	4.7	4.3
55.0		3.5	--	4.1	4.7	4.1
Avg		4.1	5.1	4.2	4.8	4.5

(a) Omitted from average because of uncertainty due to short exposure.

TABLE 2. DECOMPOSITION OF HYDRAZINE-24 HYDRAZINE-1 H<sub>2</sub>O  
NITRATE BY RADIATION AND CORROSION

Total Radia- tion Mrads	Time in Cap- sules, hr	Alloy Type				
		1100 A1	6061 A1	347 SS	Ti-6Al-4V	
<u>A. Total Gas Buildup,</u> <u>Mole x 10<sup>6</sup> per Gram of Liquid</u>						
0.5	71	18.7	17.6	54.4	23.5	
1.6	73	23.4	27.5	66.0	32.2	
11.7	96	109.8	114.0	170.0	114.0	
22.8	148	206.3	222.3	302.5	201.6	
33.4	172	285.5	310.1	433.5	279.2	
44.5	288	373.8	405.0	571.3	366.0	
55.0	312	456.5	500.6	706.5	442.2	
<u>B. Gas Buildup With Time After Exposure</u> <u>to Radiation Dose Indicated</u>						
11.7	24	7.0	4.6	24.0	8.0	
33.4	66	10.2	16.8	90.5	13.8	
55.0	144	18.2	26.6	166.2	20.8	
55.0	696	79.4	63.5	728.2	77.7	
Avg/hr		0.17	0.18	1.14	0.20	
<u>C. Gas Buildup With No Irradiation</u> <u>(Corrosion Gas)</u>						
0	264	37.3	52.6	182.5	42.4	
0	980	135.5	162.0	748.0	141.5	
Avg/hr		0.14	0.18	0.72	0.15	
<u>D. Radiolytic Produced Gas</u> <u>(Total - Corrosion Gas)</u>						
					<u>Avg</u>	
0.5		8.8	4.8	3.3	12.9	7.5
1.6		13.2	14.4	13.4	21.2	15.6
11.7		95.5	96.6	90.1	98.2	95.1
22.8		183.3	195.7	164.3	175.6	179.7
33.4		258.5	279.2	267.9	248.4	263.5
44.5		327.0	353.1	273.2	312.0	316.3
55.0		405.7	444.4	373.9	383.3	401.8
<u>E. G-Values (Molecules of</u> <u>Gas/100 ev Absorbed)</u>						
0.5		17.0	9.3	6.4	25.0	14.5(a)
1.6		8.0	8.7	8.1	12.8	8.4
11.7		7.9	8.0	7.4	8.1	7.8
22.8		7.8	8.3	7.0	7.4	7.6
33.4		7.5	8.1	7.7	7.2	7.6
44.5		7.1	7.7	5.9	6.8	6.9
55.0		7.1	7.8	6.6	6.7	7.0
Avg		7.6	8.0	7.1	8.2	7.6

(a) Omitted from average because of uncertainty due to short exposure.



very high with respect to total radiation-induced decomposition. However, in the case of the hydrazine-hydrazine nitrate solution, for which the total pressure buildup is much greater, the decomposition does not decrease with time after irradiation as rapidly as it does with hydrazine. That is, the apparent postirradiation decomposition is not only greater with hydrazine-hydrazine nitrate solutions but also persists longer. Since we know very little about the mechanism of such a reaction, we cannot say anything about the influence of either total dose or dose rate on the rate or extent of the post irradiation reaction. To predict what might happen at much lower dose rates with longer exposure times--so that the same total radiation doses are absorbed--will require considerably more investigation.

Although the G-values for a decomposition of a given fuel in the different container materials are not exactly the same, the results for the two fuels fall into two rather consistent groups. In general, the rate of radiation-induced decomposition of the hydrazine-hydrazine nitrate solution is about 50-60 percent higher than the rate of neat hydrazine. This corresponds to an overall decomposition rate of about 0.003 percent per megarad for the hydrazine and about 0.005 percent per megarad for the hydrazine-hydrazine nitrate solution.

The average G-value for hydrazine in 6061 Al is slightly lower than given in Battelle's previous report (5.1 vs 5.8). The previously reported high-dose-rate irradiations were not carried to as high total doses as in the present study. It will be noted that the G-values at low total doses are generally higher than those obtained at high total doses. Furthermore, a correction was applied for the decomposition seen as a postirradiation reaction, which was not done previously and this tends to lower slightly the results. Thus, the results are quite comparable. However, the same cannot be said for the results obtained in Ti-6Al-4V vessels. Although the G-values might be slightly smaller for the reasons given above, the reason for the observed decrease cannot be explained. Perhaps the difference is due to the more rigorous cleaning procedure used in the latest experiments, but we do not really know.

It will also noted that the G-values in all cases with both fuels show a tendency to decrease with increasing doses. The reason for this trend is at present unknown. More work will be needed before these factors are understood well enough to predict performance over long periods of time.

#### Composition of Decomposition Gases

The compositions of the decomposition gases produced in the two propellants were obtained by mass spectrometric analysis of noncondensable gases at Dry-Ice temperature before and after room-temperature irradiation in glass vessels with and without metal specimens. The results of these analyses are presented in Table 3. As these experiments were conducted in glass vessels, reactions between propellant and specimens could be evaluated periodically for corrosion-gas generation. In the case of  $N_2H_4$  there was no evidence of reaction, while with the  $N_2H_4-24N_2H_5NO_3$  gas bubbles evolved strongly from 6061 Al, a few from 1100 Al and 347 SS, and none evolved from the Ti-6Al-4V specimens that were immersed in the liquid. This gassing initiated during the first 17-hour exposure to the radiation field. However, during room storage, gassing not only did not start immediately, but was of much lower magnitude than that in the irradiated vessel, even after 2 months storage. The composition of the gas over  $N_2H_4$  was essentially unchanged by the presence of metal specimens. In the case of  $N_2H_4-24N_2H_5NO_3$ , the presence of metals appeared to increase the  $N_2O$  concentration in the non-condensable gases. As indicated in Sections B and C of Table 2 and discussed later in this report, there is a significant reaction between this propellant and the various alloys (particularly, the 347 SS).

#### Compatibility of Container and Feed-System Materials with Propellants

Several types of exposure were employed in the evaluation of the compatibility of propellants with the potential container materials and feed-system components:

TABLE 3. COMPOSITION OF NONCONDENSIBLE GASES OVER PROPELLANTS  
BEFORE AND AFTER EXPOSURE TO GAMMA RADIATION

Propellant (Specimens)	Dose Mrads	Collection Tempera- ture, °C	Volume Percent of Gases					
			N <sub>2</sub>	H <sub>2</sub>	NH <sub>2</sub>	N <sub>2</sub> O	AR	N <sub>2</sub> H <sub>4</sub>
N <sub>2</sub> H <sub>4</sub>	0	25	0	trace	90+	0	0	7
N <sub>2</sub> H <sub>4</sub>	51	-78	49	43	8	0	0	0
N <sub>2</sub> H <sub>4</sub> (plus two specimens each of four alloys)	51	-78	52	43	4	0	1	0
N <sub>2</sub> H <sub>4</sub> - 24N <sub>2</sub> H <sub>5</sub> NO <sub>3</sub> - 1 H <sub>2</sub> O	51	-78	84.3	14.4	0.02	1.2	0.08	0
N <sub>2</sub> H <sub>4</sub> - 24N <sub>2</sub> H <sub>5</sub> NO <sub>3</sub> (plus two specimens of four alloys)	75	-78	81.0	14.3	0.2	4.5	0	0

- (1) Stress (U-Bend) Specimens in Propellants
  - (a) Room storage
  - (b) Exposed to  $\text{Co}^{60}$  source of 0.52 megarad/hour for 50-70 hours
  - (c) Exposed to  $\text{Co}^{60}$  source of 0.004 megarad/hour for 1500 hours
- (2) Propellants Contained in Metal Capsules
  - (a) Room storage
  - (b) Exposed to  $\text{Co}^{60}$  source of 0.52 megarad/hour for 105 hours
  - (c) Postexposure storage
- (3) Tubing, Filter, and O-Rings in Propellants
  - (a) Room storage
  - (b) Exposed to  $\text{Co}^{60}$  source for  $\sim 100$  hours.

#### Propellants and Stressed Specimens

Specimens of the four alloys were exposed to the propellants in Pyrex vessels for periods of up to 2 months in and out of a radiation field and then were examined by optical metallographic methods and by electron diffraction to determine what changes in surface structure occurred as a result of these exposures. In all cases, specimens were exposed to both the liquid and the vapors of the propellant (see Figure 1). When exposed to the liquid (in room storage) all four alloys remained bright with no evidence of a deposit or growth of reaction product. However, some very thin stains were observed to form on specimens exposed (in room storage only) to the propellant vapors for extended periods of time ( $>1$  month). The stains were a light amber color and probably did not exceed 1000 Å in thickness. These stains were visible at low or no magnification with oblique illumination but were not detectable with vertical illumination at high (200-400X) magnifications. The specimens exposed to liquid  $\text{N}_2\text{H}_4 \cdot 24\text{N}_2\text{H}_5\text{NO}_3$  for approximately 3 weeks were observed to evolve gas bubbles throughout the exposure period. After 4 to 5 weeks' exposure, the clear liquid had turned light pink and darkened with exposure. This discoloration has been attributed to the formation and dissolution of  $\text{Fe}(\text{NO}_3)_3$  in the nitrate-containing propellant, as this discoloration occurred only in the 347 SS capsules with nitrate propellant. No discoloration was observed in any of

the compatibility studies with  $N_2H_4$  in or out of the gamma radiation field. The colorless nitrate propellant turned to light amber in a glass vessel when exposed to the radiation field for 3 to 5 days if the metal samples were included but remained colorless in their absence. Thus, radiation appears to accelerate the nitrate propellant reaction with the specimens. Although gas evolution and propellant discoloration substantiated reaction with the specimens, metallographic examinations revealed no significant changes or evidence of stress corrosion in the specimen except for a slight increase in definition of grain structure, which is indicative of a light etching by the propellants.

As invisible films are more readily detected by electron diffraction, this technique was employed to evaluate changes in surface films on the various alloys during exposure to the propellants. The sensitivity of this technique is such that air-formed films  $40 \text{ \AA}$  thick are detectable and frequently prevent diffraction from the substrate. Hence, the results presented in Table 4 for exposed specimens must be compared with those obtained from specimens treated as previously described and stored in air-filled glass vials. This prior treatment plus air storage left a very thin (probably of the order of  $50 \text{ \AA}$ ) film of hydrous oxides on Al- and Fe-base specimens and of  $TiO_2$  on the Ti-6Al-4V alloy. The film on 6061 Al was essentially unchanged by exposure to either propellant. However, the 1100 Al was etched by both propellants, which appear to remove the hydrous oxide film and leave the inclusion phase, MnS, standing in relief on the surface. Both propellants cleaned the Ti-6Al-4V specimens but left TiO particles on the surface. In the case of 347 SS, the  $N_2H_4$  slightly altered the normal hydrous oxide film, but the  $N_2H_4$ - $24N_2H_5NO_3$  produced metal oxide that undoubtedly contains some Ni and/or Cr substituted for Fe ions, as pure FeO neither forms nor is stable at room temperature. Although the magnitude of the reactions in these short-time exposures was not sufficient to produce visible indication, the reactions may be important factors in 10-year missions. It appears that the behavior of 347 SS in  $N_2H_4$ - $24N_2H_5NO_3$  would introduce more difficulties than that of the other material from the standpoint of fuel contamination, reaction, and gas evolution. As will be seen in a later section of this report, feed-system tubing of 347 SS may plug up when exposed to this propellant.

TABLE 4. RESULTS OF ELECTRON DIFFRACTION STUDIES ON SPECIMENS EXPOSED TO THE PROPELLANTS

Exposure Conditions	Films Identified on			
	1100 Al	6061 Al	347 SS	Ti 6-4
(1) Chemically cleaned, stored in glass vial	Al+ChiAl <sub>2</sub> O <sub>3</sub> H <sub>2</sub> O	Al+EtaAl <sub>2</sub> O <sub>3</sub> H <sub>2</sub> O	Fe(OH) <sub>3</sub>	TiO <sub>2</sub> (Rutile + Anatase)
(2) Chemically cleaned, N <sub>2</sub> H <sub>4</sub> vapor	Al+MnS	Ditto	FeO(OH)	TiO + Alpha Ti
(3) Chemically cleaned, N <sub>2</sub> H <sub>4</sub> liquid	Al+MnS	Al <sub>2</sub> O <sub>3</sub> H <sub>2</sub> O Boehmite	Ditto	Ditto
(4) As (2) irradiated 5 days	Al+MnS	Al+EtaAl <sub>2</sub> O <sub>3</sub> H <sub>2</sub> O	"	"
(5) As (2) irradiated 2 months	ChiAl <sub>2</sub> O <sub>3</sub> H <sub>2</sub> O	Ditto	"	"
(6) As (3) irradiated 5 days	Al+MnS	"	"	"
(7) As (3) irradiated 2 months	Al+MnS	"	"	"
(8) Chemically cleaned, N <sub>2</sub> H <sub>4</sub> -24%N <sub>2</sub> H <sub>5</sub> NO <sub>3</sub> vapor	Al+MnS	Al <sub>2</sub> O <sub>3</sub> H <sub>2</sub> O Boehmite	MO(a)	TiO <sub>2</sub> (Rutile + Anatase)
(9) Chemically cleaned, N <sub>2</sub> H <sub>4</sub> -24%N <sub>2</sub> H <sub>5</sub> NO <sub>3</sub> liquid	Al+MnS	Al+EtaAl <sub>2</sub> O <sub>3</sub> H <sub>2</sub> O	Ditto	TiO + Alpha Ti
(10) As (8) irradiated 5 days	MnS+ChiAl <sub>2</sub> O <sub>3</sub> H <sub>2</sub> O	Ditto	"	Ditto
(11) As (8) irradiated 2 months	Al+ChiAl <sub>2</sub> O <sub>3</sub> H <sub>2</sub> O	"	"	"
(12) As (9) irradiated 5 days	Al+MnS	"	"	"
(13) As (9) irradiated 2 months	Al+ChiAl <sub>2</sub> O <sub>3</sub> H <sub>2</sub> O	"	"	"

(a) This MO phase is the monoxide of Fe, N and undoubtedly contains both ions and possible Cr ion.

### Propellants in Capsules

The four alloys, in the form of capsules, were exposed to the propellants under room storage for  $\sim 1000$  hours and in a radiation field of 0.52 megarad/hour for 105 hours to determine which of the alloys would provide stable containers for the propellants. The compatibility of each combination was initially evaluated by the increase in the gas pressure over the liquid propellant as a function of storage time before and after an exposure to gamma radiation. These results are presented in Sections B and C of Tables 1 and 2. There was little or no evidence from gas-evolution data of reaction between  $N_2H_4$  and any of the capsule materials in the absence of radiation and only slight evidence of reaction after irradiation. The amount of gas generated did not appear to increase with time of storage. In contrast with these results, the gas-evolution data in capsules containing  $N_2H_2-24N_2H_5NO_3$  indicated an appreciable, continuous reaction in both irradiated and nonirradiated conditions. With the 347 SS capsule, the rate of corrosion-gas generation was 5 to 8 times that of the other capsule materials containing the  $N_2H_4-24N_2H_5NO_3$  propellant.

Upon completion of the gas-evolution studies, the capsules were opened and the remaining liquid was removed. The capsules were examined metallographically for evidence of surface degradation. As with the coupons, the extent of surface reaction was not sufficient to be detected by optical examination. However, very thin films as were observed by electron diffraction on the coupon test specimen, undoubtedly formed on the capsule surfaces. Time did not permit an examination of each of these capsules by electron diffraction. The results of visual examination of the liquid and the capsule walls were consistent with the gas-evolution results. With  $N_2H_4$  the liquid remained clear but developed a slight amber color in some cases and no significant change occurred on the capsule walls except in the case of the 347 SS capsule on which the surfaces were brighter after exposure to  $N_2H_4$  than in the as-made and treated condition. A marked reaction was evident between 347 SS and the nitrate-containing propellant as shown by

the dark red-violet color of the remaining liquid and the brownish discoloration of the capsule surfaces. This film was too thin to be extracted for X-ray diffraction analysis but, based upon the electron-diffraction examination of the coupon specimen it is believed to be MO where M = Fe, Ni, or Cr. This deposit is probably responsible for the initial plugging of the 347 SS capillary tubing exposed to the nitrate propellant. The Ti-6Al-4V capsule showed the least change in surface structure when exposed to  $\text{N}_2\text{H}_4\text{-}^{24}\text{N}_2\text{H}_5\text{NO}_3$ . The Al-alloy capsule exhibited a slightly matte-type surface indicating some etching by the propellant. Perhaps this etch is responsible for the slight amber-smoke color of the liquid extracted from these capsules. The used propellant was not analyzed for metal-ion pickup.

#### Propellants and Feed-System Components

The effect of propellant exposure on the capabilities of feed-system components was evaluated by the functional operation of the components after exposure in room storage as well as in a radiation field. Propellant flow rates, through 24-inches of 10-mil-ID tubing coiled to 1/2-inch diameter were determined before and after 119-hour exposure to a radiation dose of 60 megarads. The results tabulated below show no significant plugging after exposure to either propellant. However, with the  $\text{N}_2\text{H}_5\text{NO}_3$  containing propellant the flow stopped in the initial run after a room storage of 5.04 hours but, upon cleaning, the flow increased to more than the original value. Perhaps a slight etch by the propellant opened the tube bore.

<u>Propellant</u>	<u>Flow Rate, cc/sec</u>	
	<u>Before</u>	<u>After</u>
Hydrazine at 80 psig	0.083 ± 0.001	0.080 ± 0.001
Hydrazine/hydrazine nitrate at 85 psig	0.059 ± 0.001	0.061 ± 0.001

The commercial filter was disassembled prior to use and the parts were examined microscopically, revealing several types of contamination in the channels of the wafers. This contamination consisted of (1) an adherent



stain that appeared to have been produced by the chemical-etch preparation of the channels, (2) brown chunks of glassy material (probably photoresist), and (3) miscellaneous particles. In view of these observations, the entire wafer assembly was cleaned by alcohol washings and each wafer was inspected before reassembling. The chemical stains remained on some wafers, which were placed at the top of the stack for ease of locating after use. There was no measurable resistance to flow of either propellant through the filters for the quantities ( $\sim 50$  cc) used. After exposure of the filter in hydrazine to 60 megarads of radiation, there was no change in flow characteristics and a microscopic examination of the wafers revealed no change in either the stained or clean wafers.

The filter was reassembled, filled with hydrazine/hydrazine nitrate, and irradiated while immersed in the propellant. Again, no measurable change in flow characteristics of the filter could be detected. Metallographic examination revealed slight staining of the wafer during exposure to nitrate but no distinct buildup of corrosion products that would interfere with operation of the filter. Visual examination of the other parts of the filter revealed no marked attack or changes attributed to the propellants or radiation except for the O-ring seal.

Although the drawing supplied with the filter specifies a butyl O-ring, the one incorporated in the filter was made from ethylene-propylene. An investigation was made of the compatibility of both of these materials with the propellants in and out of the radiation field. The butyl O-rings softened, distorted, and reacted with the propellants. While the ethylene-propylene O-ring distorted in the radiation field to conform to its retaining cavity and did not return to its original shape, it did not appear to soften or react with the propellants.

Effects of radiation, propellants, and stress on flat stock of ethylene-propylene were evaluated to differentiate these factors in the operation of seals or diaphragms constructed of this material. Flat and folded specimens were exposed to radiation and to room storage in and out of the propellants. No measurable effects were observed on the flats by any treatment or on the folded specimen in room storage. However, all folded specimens took a permanent set when exposed to gamma radiation for

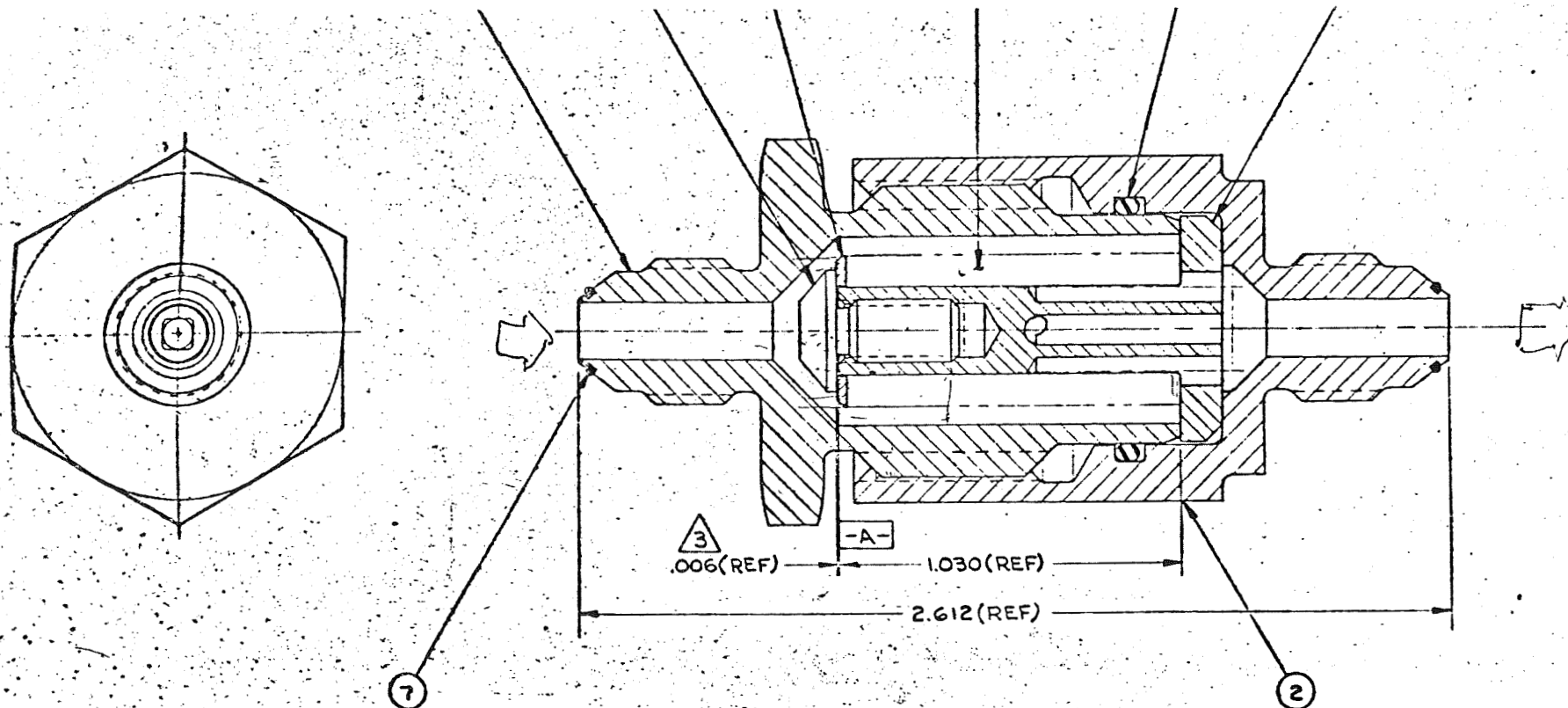
a dose of 33.4 megarads. Slight welding occurred where the folds contacted. Since both polyethylene and polypropylene are well known to crosslink by radiation, these results are to be expected. Further investigation of this material was beyond the scope of this program.

Examination of Containers From Prior  
Fuel/Oxidizer Radiation-Effects Studies

Capsules of 316 SS, Ti6Al-4V, and 1100 Al, which had been employed in radiation-stability studies of the fuels  $B_2H_6$ ,  $C_3H_8$ , and  $N_2H_4$  or oxidizers  $O_2F_2$  and  $N_2O_4$  were sectioned and examined for surface reactions that could interfere with operation of propellant systems designed for long-term space missions. Details of the radiation exposures are given in Document CR109767. Unfortunately, capsules exposed to these fuels and oxidizers without radiation were reused in some of the previous radiation-effects experiments, so no comparisons can be made of corrosion effects in and out of radiation fields. Therefore, the results in Table 5 (obtained in the present study) are applicable only to systems exposed to the respective environment in a radiation field. These observations indicate that, in some cases, oxidizers and fuels do react with the various materials and thus change their surface characteristics. The extent of reaction was most significant with the stainless steel and least significant with the titanium. With the aluminum, the reaction was spotty. Some discoloration and particle-type debris were undoubtedly a result of fabriacting or sectioning the capsules, particularly in the case of the aluminum capsule, as metallic Al was identified by X-ray diffraction examination of large particles adhering to the wall of the capsule. Metallographic examination of cross sections of these capsules revealed no pitting or in-depth attack.

RADIATION EFFECTS WITH VARIOUS FUELS AND CONTAINERS

Capsule Material	Exposure Environment	Results of Visual and X-ray Diffraction Examination
SS	$B_2H_6$	Deposit of $B_2O_3$ + unidentified iridescent crystals up to liquid level in capsule. Above liquid no apparent reaction.
SS	$O_2F_2$	Orange-brown particles identified as $CrF_3H_2O$ + $NiF_2$ + unidentified phase in bottom with a few spots on side walls.
SS	$N_2O_4$ inhibited	Deposit of yellow particles (unidentified) in bottom. Side walls clean.
SS	$N_2O_4$	Brown stain over bottom and walls of capsule with tan crystals $Fe(OH)_2$ deposited over stain.
Al	$N_2H_4$	Oily residue in capsule which reacted with air or dried to $\alpha Al_2O(OH)_2$ plus one unidentified line.
Al	$C_3H_8$	Bottom moist - sides dry, spot of brown and white corrosion which have not been identified.
Ti	$C_3H_8$	No evidence of corrosion or deposits.
Ti	$N_2H_4$	Oily residue in bottom of capsule which left a very thin (water drop type) stain upon standing.



- 3 NUMBER OF FILTER ELEMENTS (2) TO BE DETERMINED ON ASSEMBLY TO LOCATE RING (5) FACE WITHIN .006 IN. OF SURFACE -A- OF RETAINER (3) AS SHOWN. SEE DRAWING D7116970 FOR TOOLING.
- 1, 6, 7 LUBRICATE THREADS OF (4) & (1) & O-RING (8) WITH "APIEZON L" PRIOR TO ASSEMBLY.
- 8 REMOVE ALL BURRS AND SHARP EDGES.

FIGURE 4. FUEL FEED-SYSTEM FILTER

QWB PART NO.	OR NO.	DASH NO.	DESCRIPTION	SPECIFICATION	STOCK SIZE REF DESIGNATION: ELEC CH2	MATERIAL	QTY REQD	QTY
D90568-017-1009			O'RING		.676 NOM I.D. X .070	BUTYL	1	8
D90163-4-1006			O'RING		.186 NOM ID X .025	BUTYL SHORE 70	2	7
D125898-2			FILTER ELEMENT				A/R	4
C125900			RING, RETAINER				1	5
C9116969			SCREW, RETAINER				1	4
D9116968			RETAINER, FILTER				1	3
D9116967			FITTING, OUTLET				1	2
D9116966			FITTING, INLET				1	1

QWB PART NO.	OR NO.	DASH NO.	DESCRIPTION	SPECIFICATION	STOCK SIZE REF DESIGNATION: ELEC CH2	MATERIAL	QTY REQD	QTY
			ALPS TEST					
			USED ON					

QWB PART NO.	OR NO.	DASH NO.	DESCRIPTION	SPECIFICATION	STOCK SIZE REF DESIGNATION: ELEC CH2	MATERIAL	QTY REQD	QTY
			ALPS TEST					
			USED ON					

QWB PART NO.	OR NO.	DASH NO.	DESCRIPTION	SPECIFICATION	STOCK SIZE REF DESIGNATION: ELEC CH2	MATERIAL	QTY REQD	QTY
			ALPS TEST					
			USED ON					

SUMMARY

The results of these investigations show hydrazine to be compatible with all four commercial alloys, with only small variations among the alloys. These variations are not understood but may indicate small differences in chemical reactivity. The hydrazine-24 hydrazine nitrate reacted differently with each of the alloys and reacted most with the 347 SS. Although this did not result in deposits forming on the steel, the propellant discolored appreciably, indicating dissolution (etching) by the propellant. Metallographic examinations indicated a similar reaction with all of the alloys. It is suggested that chemical assays for metal content of the propellants after exposure to the various materials are needed to determine the rate of attack. However, it is expected that longer exposures will be needed to establish an equilibrium rate before a reasonable extrapolation to 10-year life can be made. Even though the observations show the stainless steel to be most reactive, the other alloys produced significant quantities of corrosion gas, and their compatibility with the nitrate-containing propellant is questionable. Furthermore, the role of radiation in effecting the initiation of this attack needs additional clarification in regard to dose rates and total dosage effects. Although differences between stressed and unstressed specimens were detected, physical-property changes were not evaluated.

Two findings appear to be important in the radiolytic decomposition of these propellants: (1) the initial rate is significantly larger than that observed as the total dose is increased and (2) the final rate does not appear to have reached a steady state. It has not been established whether these factors are time or dose-rate dependent. However, the corrosion cannot be ignored, as it could cause this type of behavior to be exhibited. As the thin surface films detected by electron diffraction cover the surface, the reactivity with the propellants would be expected to be lower. Perhaps treating the surfaces of containers or fuel-system components with passivators prior to use would reduce the corrosion factor so that the radiolytic-decomposition factors could be evaluated more precisely.

It is evident from the examination of the compatibility of the butyl and ethylene-propylene materials with the propellants and with gamma radiation that their properties can be altered by exposure to the environments expected on the space-probe mission. Details of these changes or their ultimate effect on operation of the mission have not been considered in this program.

APPENDIX A

PREPARATION OF  $\text{N}_2\text{H}_4$   $\text{HNO}_3$ - $\text{N}_2\text{H}_4$  MIXTURE

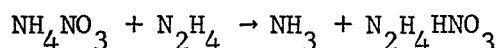
## APPENDIX

PREPARATION OF  $N_2H_4 \cdot HNO_3$ - $N_2H_4$  MIXTURE

by

Stephen P. Vango

Figure A-1 shows the setup for preparing a mixture of hydrazine nitrate dissolved in hydrazine. The required quantity of ammonium nitrate is added to anhydrous hydrazine to give the desired concentration of hydrazine nitrate. The equation for this preparation is



To prepare a mixture containing 24 percent of hydrazine nitrate in hydrazine, 0.2403 g of ammonium nitrate is added per gram of hydrazine. No more than 800 ml of hydrazine is placed in the 1-liter flask (see Figure A-1). The procedure is as follows:

(1) The three-neck flask is loaded with weighed anhydrous hydrazine and the 250-ml flask is loaded with the weight of ammonium nitrate necessary to give the desired quantity of hydrazine nitrate dissolved in hydrazine. The joints are lubricated only at the upper portion with Apiezon N grease so as to minimize contact between the grease and the hydrazine. The stopcock on the Dewar-type trap is sparingly lubricated with Apiezon N.

(2) The Dewar-type trap is filled with Dry Ice and the loaded system is evacuated through a liquid-nitrogen trap ahead of the pump. The Dry Ice prevents the loss of hydrazine but permits the  $NH_3$  to be pumped off.

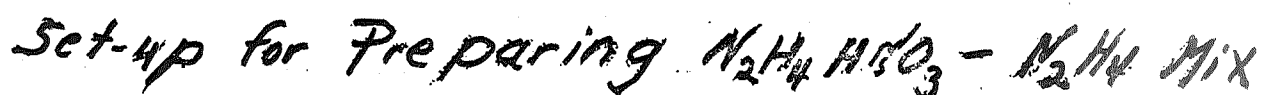
(3) The 250-ml flask is rotated on its joint and the tubulation is tapped with a rubber stopper attached to a glass rod to cause the ammonium nitrate to drop into the stirred hydrazine. The liberated  $NH_3$  is pumped off. Care must be taken to add the ammonium nitrate in small portions so as not to cause too violent an evolution of  $NH_3$ . If the evolution becomes violent, the partially open stopcock should be closed momentarily.

DET-661862



(4) When all the ammonium nitrate has been added, the stopcock is closed and the assembly is tipped carefully so as to introduce the solution into the 250-ml flask to rinse it of all ammonium nitrate.

(5) With no undissolved ammonium nitrate in evidence, the stirred solution is pumped for about 30 minutes or until the solution no longer bubbles from the interior and the liquid-nitrogen trap ahead of the pump has quieted down, indicating no more condensation of  $\text{NH}_3$ .



S.P. Yang  
11/10/1969

Fig. A-1

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